
मेथनॉल की विशिष्टि (मेंथाइल अल्कोहल)
(तीसरा पुनरीक्षण)

Specification for Methanol
(Methyl Alcohol)
(Third Revision)

ICS 71.080.60

© BIS 2020



भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first issued in 1954 stipulating the requirements and methods of test for three grades of methanol, namely, Grade 1, Grade 2 and Special. In the first revision, two grades, namely, methanol pure and technical were covered, the requirements of the special grade, mainly intended for defence purposes were suitably covered under those of methanol, pure. High purity methanol required for the manufacture of antibiotics was also taken care of by prescribing additional requirements. Improvements in the test for alkalinity, a simpler alternative method for determination of sulphur and sulphur compounds and a revised scheme of sampling and criteria for conformity, were some of the salient features of this revision.

In drawing up second revision, the basic consideration was to stipulate the requirements on the basis of authentic data on the indigenously produced material and to incorporate all other changes found necessary as a result of experience gained through the use of the original standard. In order to take care of the some of the important users of methanol, second revision was undertaken in which, the requirements for specific gravity and distillation range were modified accordingly.

The Committee, responsible for revision of the standard, has observed that, now a days more sophisticated instruments having improved minimum detection limits are available for testing iron, aldehyde and ketone. Considering this, the committee decided to revise the standard. In this revision (third) alternate test methods for iron, aldehyde and ketone have been added and Amendment no. 1 and 2 have also been incorporated in the standard.

The composition of the Committee, responsible for the formulation of this standard is given at Annex H.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SPECIFICATION FOR METHANOL (METHYL ALCOHOL)

(*Third Revision*)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for methanol (methyl alcohol).

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on these standards are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
82 : 1973	Methods of sampling and test for thinners and solvents for paints (<i>first revision</i>)
170 : 2004	Acetone — Specification (<i>fourth revision</i>)
264 : 2005	Nitric acid — Specification (<i>third revision</i>)
265 : 1993	Hydrochloric acid — Specification (<i>fourth revision</i>)
321 : 1964	Specification for absolute alcohol (<i>revised</i>)
323 : 2009	Rectified spirit for industrial use — Specification (<i>second revision</i>)
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
1448 (Part 18) : 1991	Methods of test for petroleum and its products: Part 18 Distillation of petroleum products (<i>second revision</i>)
2362 : 1993	Determination of water by Karl fischer method — Test method (<i>second revision</i>)

*IS No.**Title*

2263 : 1979	Methods of preparation of indicator solutions (<i>first revision</i>)
4905 : 2015	Random sampling and randomization procedures (<i>first revision</i>)

3 GRADE

There shall be two grades of the material, namely:

- a) Methanol, pure; and
- b) Methanol, technical.

4 REQUIREMENTS**4.1 Description**

The material shall be a clear and colourless liquid, free from matter in suspension.

4.2 Miscibility with Water

The material shall show no turbidity when one volume is mixed with 19 volumes of water at 27 ± 2 °C and examined as prescribed in Annex A.

4.3 The material shall also comply with the requirements given in Table 1.

4.4 Methanol for Manufacture of Antibiotics

It shall, in general, comply with the requirements for methanol, pure, given in Table 1, except for the characteristics given in Table 2, for which separate requirements have been prescribed.

5 PACKING AND MARKING**5.1 Packing**

5.1.1 The material shall be packed in containers as agreed between the purchaser and the supplier, subject to the provisions of the law in force in the country for the time being.

Table 1 Requirements for Methanol
(Clauses 4.3, 4.4 and 7.1)

Sl No.	Characteristic	Requirements For Grade		Method of Test, Ref to	
		Methanol Pure	Methanol Technical	Indian Standards	Annex
(1)	(2)	(3)	(4)	(5)	(6)
i)	Relative density at 27/27 °C, <i>Max</i>	0.789	0.798	6 of IS : 82	-
ii)	Distillation range at 760 mm pressure	The difference between initial boiling point (IBP) and dry point (DP) shall not exceed 1 °C including 64.5 °C	Not less than 95 percent by volume shall distill between 65.5 °C and 66.5 °C [temperature being corrected for a pressure of 760 Hg] (<i>see</i> Note below)	(Part 18) of IS 1448	-
iii)	Residue on evaporation, percent by mass, <i>Max</i>	0.01	0.015	8 of IS 82	-
iv)	Alkalinity (as NH ₃), percent by mass, <i>Max</i>	0.03	0.0003	-	B-1
v)	Acidity as acetic acid (CH ₃ COOH), percent by mass, <i>Max</i>	0.004	0.006	-	B-2
vi)	Aldehydes and ketones as acetone (CH ₃ COCH ₃), percent by mass, <i>Max</i>	0.03	0.100	-	C-1 or C-3
vii)	Sulphur and compounds of sulphur (as S), percent by mass, <i>Max</i>	0.001	-	-	D
viii)	Chlorine and chlorine compounds	To pass the test	-	-	E
ix)	Water content, percent by mass, <i>Max</i>	0.1	0.5	IS 2362	-
x)	Purity, percent by mass, <i>Min</i>	99.85	-	-	G

NOTE — In case of barometric pressure is lower than 760 mm Hg, use the correction to the observed temperature by adding 0.034 °C for every mm below 760 mm. This correction is valid for a pressure range of 700 to 760 mm of Hg

Table 2 Special Requirements for Methanol Used For Antibiotics
(Clauses 4.4 and 7.1)

(Clause 4.4)				
Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No. in	
			Indian Standard	Annex
(1)	(2)	(3)	(4)	(5)
i)	Acidity as acetic acid, percent by mass, <i>Max</i>	0.002	-	B – 2
ii)	Aldehydes and ketones as acetone (CH ₃ COCH ₃), percent by mass, <i>Max</i>	0.001	-	C – 2 or C – 3
iii)	Residue on evaporation, percent by mass, <i>Max</i>	0.001	8 of IS 82	-

5.1.2 All containers, in which the material is packed, shall be dry, clean and free from substances soluble in methanol and shall be leak proof.

5.1.3 Necessary safeguards against the risks arising from the storage and handling of large volumes of flammable liquids shall be provided and all precautions shall be taken at all times to prevent accident by fire or explosion.

5.1.4 Except when they are opened for the purposes of cleaning and rendering them free from vapour, all containers shall be kept securely closed, unless they

have been thoroughly cleaned and freed from methanol vapour.

5.2 Marking

5.2.1 The material shall be supplied in accordance with the marking and delivery instructions given by the purchaser.

5.2.2 Each container shall be marked with the:

- Name of the material;
- Manufacturer's name or trade-mark, if any, or both;

- c) Gross, net and tare mass;
- d) Month and year of manufacture; and
- e) Minimum cautionary notice worded as under;

‘FLAMMABLE! INGESTION OR ABSORPTION
DANGEROUS! CAUSES BLINDNESS!
CUMULATIVE POISON!’

5.2.3 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in Annex F.

7 TEST METHODS

7.1 Tests shall be conducted according to the methods prescribed in Annexes and various Indian Standards. References to various standards and relevant Annexes are given in col 5 and 6 of Table 1, and col 4 and 5 of Table 2.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the result of analysis.

ANNEX A

(Clause 4.2)

TEST FOR MISCIBILITY WITH WATER

A-1 PROCEDURE

A-1.1 Mix 10 ml of the material with 190 ml of distilled water in a suitable glass vessel and allow to stand at any temperature within the range 27 ± 2 °C for one hour.

Compare the clarity of the mixture with that of an equal volume of distilled water.

A-1.1.1 The material shall be taken to comply with the requirement if there is no noticeable difference in clarity between the mixture and water.

ANNEX B

[Table 1, Items (iv) and (v) and Table 2, Item (i)]

DETERMINATION OF ALKALINITY AND ACIDITY

B-1 ALKALINITY

B-1.1 Reagents

B-1.1.1 *Methyl Red Solution*, 0.15 g in 500 ml of water (see IS 2263).

B-1.1.2 *Standard Sulphuric Acid*, 0.01 N.

B-1.2 Procedure

Place 100 ml of water and a few pieces of clean porous porcelain in a 500 ml resistance glass conical flask and boil gently for 5 min to eliminate carbon dioxide. Cool with the neck of the flask closed with a stopper carrying a soda lime tube. Add to it one drop of methyl red solution and titrate to a faint pink colour with standard sulphuric acid. To this, add 100 ml of the material and titrate to the same faint pink colour with standard sulphuric acid. Calculate the alkalinity.

B-1.3 Calculation

$$\text{Alkalinity (as NH}_3\text{), percent by mass} = \frac{1.7 VN}{100S}$$

where

V = volume in ml, of standard sulphuric acid solution used in titration with material;

N = normality of standard sulphuric acid solution; and

S = specific gravity of the material at the temperature of determination.

B-2 ACIDITY

B-2.1 Reagents

B-2.1.1 *Phenolphthalein Indicator*, prepared by dissolving 0.1 g of phenolphthalein in 100 ml of 60 percent ethyl alcohol (see IS 2263).

B-2.1.2 *Standard Sodium Hydroxide Solution*, 0.01 N.

B-2.2 Procedure

Place 100 ml of carbon dioxide free water as prepared in **B-1.2** in a 500 ml resistance glass conical flask. Cool slightly and add 100 ml of the material. Boil gently for a further period of 5 min. At the end of this period, close the neck of the flask with a stopper add 0.5 ml of phenolphthalein indicator and examine for alkalinity, if not alkaline, titrate with standard sodium hydroxide solution using a micro-burette.

B-2.3 Calculation

$$\text{Acidity (as CH}_3\text{COOH), percent by mass} = \frac{6VN}{100S}$$

where

V = volume in ml, of standard sodium hydroxide solution used;

N = normality of standard sodium hydroxide solution; and

S = specific gravity of the material at the temperature of determination.

ANNEX C

[Table 1, Item (vi) and Table 2, Item (ii)]

DETERMINATION OF ALDEHYDES AND KETONES

C-1 HYDROXY AMMONIUM CHLORIDE METHOD**C-1.0 Outline of the Method**

Reaction with hydroxylamine hydrochloride forms the basis for determination of carbonyl function (aldehydes and ketones). Condensation products called oximes are produced. The hydrochloric acid released in the reaction is titrated with standard sodium hydroxide solution using bromophenol blue as indicator.

C-1.1 Reagents

C-1.1.1 *Standard Sodium Hydroxide Solution*, 0.1N.

C-1.1.2 *Rectified Spirit*, 95 percent by volume (see IS 323).

C-1.1.3 *Bromophenol Blue Indicator*, dissolve 0.2 g of bromophenol blue in 3 ml of the standard sodium hydroxide solution and dilute to 100 ml with rectified spirit.

C-1.1.4 *Hydroxylamine Hydrochloride Solution*, dissolve 4g of hydroxyl-amine hydrochloride in 20 ml of water. Dilute this to 200 ml with rectified spirit, heat on a boiling water-bath for 30 min. Cool and add 5 ml of the bromophenol blue indicator with just sufficient sodium hydroxide solution (2N) to impart a dichroic yellow-green colour to the liquid.

C-1.2 Procedure

Measure 25 ml of the material into a 150 ml conical flask, add 25 ml of hydroxylamine hydrochloride solution and, after loosely stoppering, heat on a boiling water-bath for 10 min. Cool and titrate with standard sodium hydroxide solution until as near a match as possible is obtained with a control made by mixing 25 ml of distilled water with 25 ml of hydroxylamine hydrochloride in a similar 150 ml conical flask.

C-1.3 Calculation

Aldehydes and ketones (as acetone), Percent by mass =

$$\frac{0.023(V - 0.6)}{S}$$

where

V = volume in ml, of standard sodium hydroxide solution; and

S = specific gravity of the material taken at the temperature of the test.

NOTE — The correction 0.6 is subtracted from the number of milliliters of sodium hydroxide solution used, to allow for the effect on the indicator of different alcohol concentration in the experiment and control.

C-2 DINITROPHENYLHYDRAZINE METHOD**C-2.0 Outline of the Method**

In acid medium, the carbonyl compounds present are converted with dinitrophenylhydrazine into the corresponding 2, 4-dinitrophenyl hydra zones. In alkaline medium, these have a red colour, which is compared visually with colours produced by standard solution, or alternatively, the optical density is measured and the content of carbonyl compounds is estimated by reference to a standard curve.

C-2.1 Apparatus

C-2.1.1 *Water-Bath*, controlled at 60 °C.

C-2.1.2 *Graduated Flask*, 25 ml capacity.

C-2.1.3 *Photoelectric Absorptiometer or Spectrophotometer*, with 0.5 cm cells. Alternatively, flat bottom tubes of 20 ml capacity shall be used.

C-2.2 Reagents

C-2.2.1 *Carbonyl-Free Methanol* — Reflux 1000 ml of methanol with 5 g of 2, 4-dinitrophenylhydrazine and 5 drops of concentrated hydrochloric acid (sp gr 1.18) for 2 to 3 hours. Distil off methanol. Reject the first 100 ml and collect the next 800 ml, rejecting the remainder. If, in spite of the precautions taken, the distillate is found to be coloured, then it may be redistilled.

C-2.2.2 *Concentrated Hydrochloric Acid* — Specific gravity 1.18 (see IS 265).

C-2.2.3 *Dinitrophenyl Hydrazine Solution* — Dissolve 0.03 g of 2, 4 dinitrophenyl hydrazine in 40 ml of the carbonyl-free methanol containing 0.3 ml of concentrated hydrochloric acid (specific gravity 1.18) and dilute to the mark in a 50 ml one-mark volumetric flask with the carbonyl free methanol. Prepare this solution fresh each day.

C-2.2.4 *Potassium Hydroxide Solution, Freshly Prepared* — Dissolve 10 g of potassium hydroxide in 10 ml of water, cool and dilute to the mark in a 50 ml one-mark volumetric flask with the carbonyl-free methanol.

C-2.2.5 *Standard Acetone Solution* — Weigh 1.000 g of acetone and dilute to the mark in a one-mark 100 ml volumetric flask with carbonyl free methanol. Dilute 1.0 ml of this solution to 100 ml with the carbonyl free methanol. One ml of the diluted solution contains 0.1 mg of acetone.

C-2.3 Procedure

C-2.3.1 Instrumental Procedure

Prepare five solutions by diluting 1.0, 2.0, 4.0, 8.0 and 10.0 ml portions of the standard acetone solution to 25.0 ml with the carbonyl-free methanol. To 1.0 ml of each of the solutions thus obtained, contained in a test tube fitted with a ground glass stopper, add 1.0 ml, of the 2, 4-dinitrophenylhydrazine solution. Stopper the tube and heat for 50 min in the water-bath at 60 °C, cool, add 80 ml of the potassium hydroxide solution and after 5 to 15 min measure the optical density of each solution at a wavelength of 430 mμ using as a blank 10 ml of the carbonyl-free methanol treated in the same way. Prepare a calibration chart by plotting weights (in milligrams) of acetone against corresponding values of optical density.

Dilute 5.0 ml of the sample to 25.0 ml with the carbonyl-free methanol and transfer 1.0 ml of this solution to a test tube fitted with a ground glass stopper and add 1.0 ml of the 2, 4-dinitrophenylhydrazine solution. Stopper the tube and heat for 50 min in the water-bath at 60 °C, cool, add 8 ml of the potassium hydroxide solution and after 5 to 15 min, measure the optical density of the solution at a wavelength of 430 mμ using as a blank 1.0 ml of the carbonyl-free methanol treated in the same way. By reference to the calibration chart prepared as described above, read the acetone content (in milligrams) of the solution.

C-2.3.2 Visual Procedure

Dilute 5.0 ml of the sample to 25.0 ml with the carbonyl-free methanol and transfer 1.0 ml of this solution to a test tube fitted with a ground glass stopper. At the same time, prepare five solutions by diluting 1.0, 2.0, 4.0, 8.0 and 10.0 ml portions of the standard acetone solution to 25.0 ml with the carbonyl-free methanol. Transfer 1 ml of each of the solutions so obtained to a series of test tubes fitted with ground glass stoppers and treat each of the solutions in the following manner.

Add 1 ml of 2, 4-dinitrophenylhydrazine solution. Stopper the tube and heat for 50 min in the water-bath at 60 °C, cool and add 8 ml of potassium hydroxide solution.

After 5 to 15 min, compare the colour of the solution containing the sample with the series of prepared colour standards in 20 ml flat bottom tubes, noting the acetone content of the standard that most closely matches the sample solution. In the event of doubt as to which of two colours standard most closely matches the sample solution, take the mean of the two acetone contents.

C-2.4 Calculation

C-2.4.1 Instrumental Procedure

Carbonyl compounds, calculated as acetone:

Carbonyl compounds (as CH_3COCH_3), percent by

$$\text{mass} = \frac{0.5 \times M}{S}$$

where

M = mass in milligrams of acetone corresponding to the optical density recorded; and

S = specific gravity of the sample.

C-2.4.2 Visual Procedure

Carbonyl compounds, calculated as acetone:

Carbonyl compounds (as CH_3COCH_3), percent by

$$\text{mass} = \frac{0.002 \times V}{S}$$

where

V = volume in ml, of the standard acetone solution used in preparing the colour standard that most closely matches the sample; and

S = specific gravity of the sample.

C-3 GAS CHROMATOGRAPHIC METHOD

C-3.0 Outline of the Method

The test method is related to determination of the impurities namely, ethanol, acetone and ethyl methyl ketone between 0 and 100 ppm in pure methanol by gas chromatography on GC having FID detector and capillary column.

C-3.1 Apparatus

C-3.1.1 Gas Chromatograph

Any gas liquid chromatograph equipped with on column injector and FID detector can be used with following accessories and operating conditions.

Column	:	Capillary column
Column coating material/ stationary phase	:	100 Percent dimethylpolysiloxane (DB1 or equivalent)
Column length	:	60 m
Film thickness	:	5 micron
I.D.	:	0.53 mm
Carrier gas	:	Nitrogen
Syringe	:	5 microliters
Sample size	:	1 microliter
Data integration	:	Suitable GC software/ integrating device

C-3.1.2 Typical Instrument Condition

Detector	:	FID
Oven temperature	:	200 °C
Injector, temperature	:	150 °C
Detector, temperature	:	250 °C
Initial temperature	:	40 °C
Initial hold	:	10 min
Ramp-1	:	5 °C/min
Final temperature	:	200 °C
Total run time	:	45 min
Make up flow	:	N ₂ , 30 ml/min
Split flow	:	N ₂ , 35 ml/min
Septum purge flow	:	N ₂ , 8 ml/min

NOTE — The above gas chromatographic conditions are suggestive. However any GC having different columns (packed/Capillary having different length/diameter/film thickness) and different carrier gas (He, H₂ or N₂), with different calibration technique (internal standard, external standard, area normalization) may be used provided standardization/calibrations are done after setting up chromatographic conditions for required resolution.

C-3.1.3 Recorder

Relevant GC software/integrating device.

C-3.1.4 Integrating Device

Any device capable of integrating chromatograph peak areas with a repeatability of ± 1 percent or less.

C-3.1.5 Reagent and Material

- Ethanol (99 percent pure);
- Methanol, ethanol free;
- Acetone (AR grade); and
- Ethyl methyl ketone (AR grade).

C-3.1.6 Identification and Calibration**C-3.1.6.1 Identification**

Determine the retention time of each component by injecting small amount of highly pure material either individually or synthetic blend mixture.

C-3.1.6.2 Accurately prepare standard mixture containing ethanol, acetone and ethyl methyl ketone 10 ppm each in AR grade methanol. Inject with the help of a clean and rinsed glass micro syringe, 1 micro litre of standard in the column taking care that no air bubble is trapped in the syringe. Inject the standard at least twice or till the repeatable results are obtained. Carry out the calibration by external standard method. Area of each component are to be measured with suitable integrator.

C-3.1.6.3 Calculation of response factor

Divide value of std. Ethanol in ppm by value of area obtained of ethanol and take average concentration per unit area as factor.

$$\text{R.F. of ethanol} = \frac{\text{Concentration of standard ethanol}}{\text{Area of standard ethanol obtained}}$$

Same way response factor of acetone and ethyl methyl ketone are derived.

C-3.1.6.4 Analysis procedure

1 micro litre of sample is injected without any air bubble trapped in the syringe. Order of elution and their approximate retention time of other impurities are as under.

Sequence of elution	Approx Retention time in minutes with given column
Methanol	: 9.2
Ethanol	: 12.8
Acetone	: 15.5
Ethyl methyl ketone	: 22.6

C-3.1.6.5 Calculation

Concentration of ethanol in ppm = R.F. of ethanol \times mean area of ethanol in sample
 Concentration of acetone in ppm = R.F. of acetone \times mean area of acetone in sample

Concentration of EMK in ppm = R.F. of \times mean area of ethanol in sample

Total aldehyde and ketone in ppm = Concentration of acetone + Concentration of EMK

NOTE — GC method suggested as alternate method.

ANNEX D

[Table 1, Item (vii)]

DETERMINATION OF SULPHUR AND COMPOUNDS OF SULPHUR

D-0 GENERAL

Two alternative methods (D-1 and D-2) for determination of sulphur and compounds of sulphur have been prescribed in this Annex. The choice between these, shall be subject to agreement, except that while stating the requirements of the material against this characteristic, the method of determination used shall be clearly stated.

D-1 COMBUSTION METHOD

D-1.0 Outline of the Method

A known quantity of the material is burnt in a lamp. The products of combustion being drawn through sodium peroxide solution and the resulting sulphate being determined by nephelometric method.

D-1.1 Apparatus

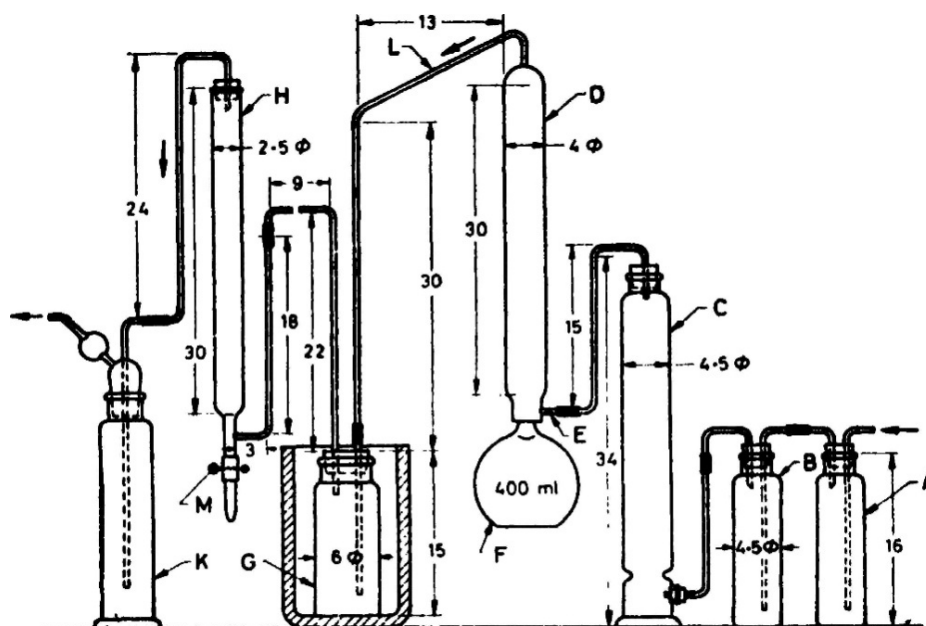
D-1.1.1 Air Purifying Train — It shall consist of two bottles A and B and a tower C, of the dimensions shown in Fig. 1, all packed with pumice. The pumice in A and C shall be soaked with potassium hydroxide solution, so that its depth in the former is 2 to 3 cm, and in the latter just sufficient to cover the inlet tube. The pumice in B shall be soaked with lead acetate solution to a depth of 2 to 3 cm.

The inlet tubes in A and B shall reach within a few millimeters of the bottom.

This arrangement removes acidic sulphur compounds only and if the air contains such compounds as carbon disulphide or thiophene, the train shall be preceded by a length of combustion tubing, packed, with coarse copper oxide, and heated to dull redness. In that case, the bottle B shall contain potassium hydroxide solution instead of lead acetate solution.

D-1.1.2 Chimney and Absorption Train — The air from the tower C shall be introduced by the side-tube E to the foot of the chimney D, into which the lamp F fits with a rubber stopper. The products of combustion shall be drawn up the chimney D and from there into the first absorption bottle G, which shall consist of a 500 ml wide-mouthed bottle, immersed in an ice-bath. The connection shall be sloped at L to avoid the possibility of condensed water running down the hot chimney. The ice-bath condenses the water and also cools the gases before they enter the sodium peroxide solution, thus avoiding undue decomposition of the latter.

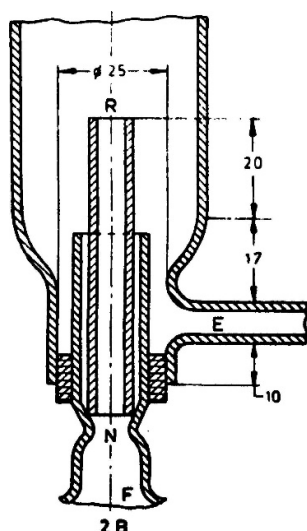
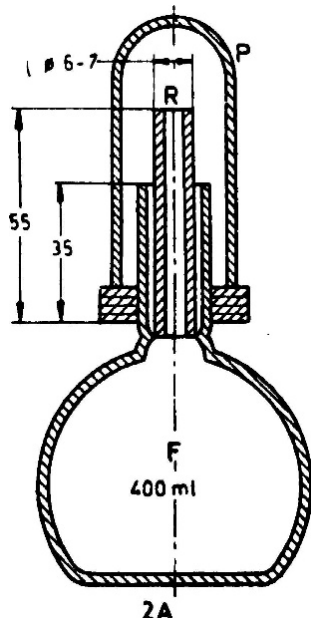
From the absorption bottle G, the gases shall pass through the tower H, containing short pieces of glass rod. The train shall be completed with the Drechsel wash-bottle K of about 250 ml capacity. The bottles G and K, and the tower H shall each contain 25 ml of sodium peroxide solution. The liquid from H can be drained by pinch-cock M.



All dimensions in Centimetres.

FIG. 1 AIR PURIFYING TRAIN

D-1.1.3 Lamp — The lamp *F* as shown in Fig. 2A shall consist of a round flask of about 400 ml capacity. The neck of the flask shall be constricted at *N* to support the tube *R* in which shall be loosely fitted an ordinary cylindrical cotton-wick. The length of *F* shall be such that when the lamp is in position, the top *R* shall be about 4 cm above the top of *E* (see Fig. 2B). During weighing, etc, a test tube *P* shall be inverted over the wick as a sheath (see Fig. 2A).



All dimensions in millimeters.

FIG. 2 LAMP

D-1.1.4 Nessler Tubes — Two tubes of thin transparent glass, about 25 mm in diameter and about 150 mm in length, graduated up to 50 ml. The depth, measured internally from the graduated mark to the bottom, shall not vary by more than 2 mm in the tubes used.

D-1.2 Regents

D-1.2.1 Absolute Alcohol, conforming to IS 321.

D-1.2.2 Potassium Hydroxide Solution, 50 percent (m/v).

D-1.2.3 Lead Acetate Solution, 10 percent (m/v).

D-1.2.4 Sodium Peroxide Solution, Dissolve cautiously 10 g of sodium peroxide in 500 ml of ice cold water.

D-1.2.5 Concentrated Hydrochloric Acid — specific gravity 1.16 (conforming to IS 265).

D-1.2.6 Barium Chloride Solution, 12 percent (m/v) of barium chloride crystals dissolved in water.

D-1.2.7 Standard Sulphate Solution, acidify 75 ml of sodium peroxide solution with concentrated hydrochloric acid, bring to boil and then cool. Transfer the solution to a 500 ml volumetric flask. Add 15.6 ml of 0.01 N standard sulphuric acid and make up the volume to 500 ml mark.

D-1.3 Procedure

D-1.3.1 Cleaning of Wick

Boil the wick for an hour with soap solution to remove oily matter and size (which might contain sulphur compounds), then boil it for several minutes with successive changes of distilled water until the water remains clear and bright. Squeeze out the wick, wash it twice in absolute alcohol, dry at $100 \pm 2^\circ\text{C}$ and insert in tube *R*.

D-1.3.2 Transfer 250 g of the material to the lamp *F*, pouring a little down the tube *R*, replace the sheath and weigh. Attach the wash-bottle *K* to a suction pump, light the lamp, draw a rapid stream of air through the apparatus and quickly push the lamp into position. The initial stream of air shall not be too slow as otherwise the lamp may be extinguished. Once the test is under way, reduce the air stream a little but take care that it is rapid enough to keep the lamp burning vigorously. When all the sample has been burned, which will take about 24 h, stop the flow of air through the apparatus, remove the lamp, replace the sheath and weigh. The difference between the two weighing, *M*, is the mass of material consumed.

D-1.3.3 Transfer contents of the vessels *G*, *H* and *K* to a beaker. If necessary, boil until the volume of the liquid is reduced to about 450 ml, then add 10 ml of concentrated hydrochloric acid. Filter and again bring it to boil. Cool the solution and make up the volume to 500 ml in a volumetric flask.

D-1.3.4 Pipette out 50 ml of the test solution (**D-1.3.3**) into a Nessler tube and add 2 ml of barium chloride

solution. Carry out a control test in another Nessler tube using 50 ml of standard sulphate solution if M is equal to 250 g (or a proportionate lower or higher volume) and 2 ml of barium chloride solution. Mix both the solutions well, stir and compare the turbidity produced, if any, after live minutes.

D-1.3.5 The material shall be considered to have satisfied the specified requirement if the turbidity produced in the test with the material is not greater than that produced in the control test.

D-2 REDUCTION METHOD

D-2.0 Outline of the Method

The sulphur present in the material is reduced with Raney nickel to metal sulphide, which is heated with hydrochloric acid. The hydrogen sulphide evolved is absorbed in sodium hydroxide solution and titrated with standardized mercuric acetate solution in the presence of dithizone as indicator. This method determines all sulphur compounds normally encountered in methyl alcohol for industrial uses. It does not, however, determine alkyl sulphur and other oxygen bounded sulphur compounds.

D-2.1 Apparatus

D-2.1.1 *Reduction Apparatus*, see Fig. 2.

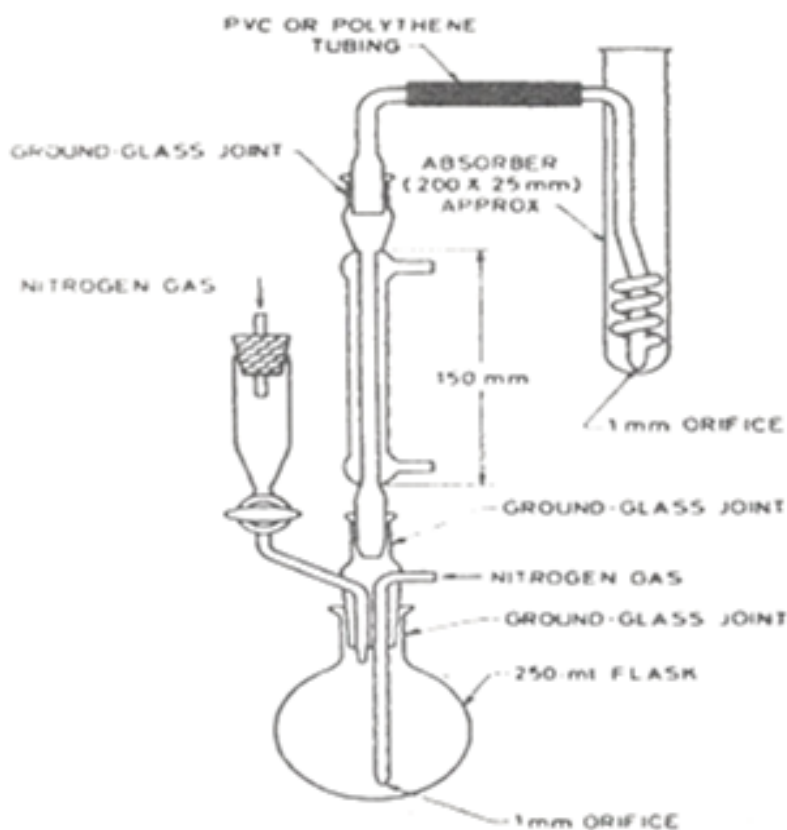


FIG. 3 REDUCTION APPARATUS FOR USE IN THE DETERMINATION OF SULPHUR AND SULPHUR COMPOUNDS

D-2.1.2 *Burette*, class A, 10 ml capacity, conforming to IS 1997 and graduated in 0.05 ml.

D-2.2 Reagents

D-2.2.1 *Nitrogen Gas*, free from carbon dioxide.

D-2.2.2 *Acetone*, conforming to IS 170.

D-2.2.3 *Raney Nickel*, treat 10 g of 50/50 nickel/aluminium alloy with 100 ml of 10 percent (m/v) sodium hydroxide solution, cooling with ice while the reaction proceeds. Wash the Raney nickel three times with water, remove the water by decantation and store the Raney nickel under 100 ml of isopropyl alcohol. Raney nickel prepared in this way retains its activity for about three days.

5 ml of this suspension will reduce quantitatively 1.0 to 1.5 mg of organic sulphur to hydrogen sulphide.

A stabilized form of Raney nickel is available commercially, and may be prepared by warming half pellet (0.24 g) in the reaction flask, in 10 ml of acetone. When the pellet has disintegrated, decant the acetone and introduce the sample.

NOTE — Raney nickel is spontaneously flammable in air.

D-2.2.4 *Sodium Hydroxide Solution*, 1.0 N.

D-2.2.5 *Hydrochloric Acid*, (Sp gr 1.18) diluted (3:2) with water.

D-2.2.6 Mercuric Acetate Solution, dissolve 0.200 g of mercuric oxide in 25 ml of water containing 1 ml glacial acetic acid, and dilute to 1000 ml with water and standardize as follows.

D-2.2.6.1 Standardization of the mercuric acetate solution, add a known amount of an aliphatic sulphur compound (di-isoamyl sulphide) to the sulphur-free methanol. Carry out a determination by the method (described in **D-2.3**) using an aliquot of this solution representing 130 to 170 µg of sulphur. From the volume of the mercuric acetate solution required in the titration, calculate the mass, in micrograms, of sulphur equivalent to 1 ml of the mercuric acetate solution.

D-2.2.7 Methanol, Sulphur-free, into a 2-litre, two-necked round bottom flask, measure 20 ml of acetone and add 0.5 g (1 pellet) of Raney nickel or 10 ml of the suspension. Warm until the pellet has disintegrated and decant the acetone. Add 1 500 ml of methanol and reflux on a water-bath. Insert the nitrogen lead in the second neck and pass in gas at the rate of 2 bubbles per second.

Heat until slight effervescence is observed on the methanol surface and continue for 30 min. Turn off the gas flow and the heating, and allow the nickel to settle. Decant the methanol into a flask and distil. Reject the first 250 ml and collect the next 1 000 ml, rejecting the remainder.

D-2.2.8 Dithizone, one percent (m/v) solution in acetone.

D-2.3 Procedure

Place 5 ml of Raney nickel suspension, or half pellet as described in the flask, then add 100 ml of the sample. Place the flask on a water-bath and pass nitrogen through at the rate of about 3 liters per hour. Adjust the temperature of the heating mantle until slight effervescence appears on the surface (just below boiling point). Continue for 50 min, swirling the flask at intervals.

Place 10 ml of the sodium hydroxide solution and 10 ml of acetone in the absorber.

Place 15 ml of the hydrochloric acid in the dropping funnel, switch off the heating mantle, and attach a second nitrogen lead to the glass tube in the top of the dropping funnel. Admit the acid, a few milliliters at a time, to the flask. Add two drops of the dithizone solution and one drop of the mercuric acetate solution to the absorber using the burette. The colour change should be from yellow to pink. Liberation of hydrogen sulphide reverses the colour change, when all the acid has been added, increase the nitrogen flow rate to about 12 liters per hour, switch on the heating mantle again, continue heating for a further 20 min, titrating with the mercuric acetate solution as necessary. When all the hydrogen sulphide has apparently been titrated,

disconnect the flow of nitrogen momentarily, cool the flask by blowing air on it and allow the absorbent to travel up the delivery tube in the absorber, to recover any hydrogen sulphide which may be absorbed on its surface.

Carry out a blank test on the Raney nickel and the sulphur-free methanol.

D-2.4 Calculation

Sulphur and sulphur compounds are calculated as:

$$S, \text{ parts per million by mass} = \frac{M \times (V_2 - V_1)}{100 \times S}$$

where

M = mass in micrograms, of sulphur equivalent to 1 ml of the mercuric acetate solution (determined as in **D-2.2.6.1**);

V_2 = volume in ml, of the mercuric acetate solution required in the test determination;

V_3 = volume in ml, of the mercuric acetate solution required in the blank test; and

S = specific gravity of the sample.

D-3 INSTRUMENTAL METHOD USING ULTRAVIOLET FLUORESCENCE TECHNIQUE

D-3.0 Scope

Total Sulfur Analyzer Based on UV Fluorescence Principle is capable for analysis of sulfur compounds having minimum detection amount 0.05 mg /lit.

D-3.1 Outline of the Method

The sample is introduced into the furnace kept at 1 000 °C. During the movement towards the furnace, sample is flushed with argon. In the furnace, sulfur components oxidize with a mixture of Ar and pure O₂ to SO₂. The gas flow travels through a perma pure membrane dryer. This device removes the water from the gas stream. After water removal, a glass fiber filter removes soot and other particles that might occur during uncontrolled combustion. The figure below explains detection principle.

Detection is based on the principle that SO₂ molecules absorb (UV) light and become excited, then decay to a lower energy state emitting UV light at a specific wavelength. The sample flows into reaction chamber, where pulse light excites the SO₂ molecules.

As the excited molecule decay to a lower energy state, an amount of UV light is emitted that is proportional to SO₂ concentration. The band-pass filter allows only the wavelengths emitted by the excited SO₂ molecule to reach the photo multiplier tube (PMT). The PMT detects UV light emission from the decaying SO₂ molecule.

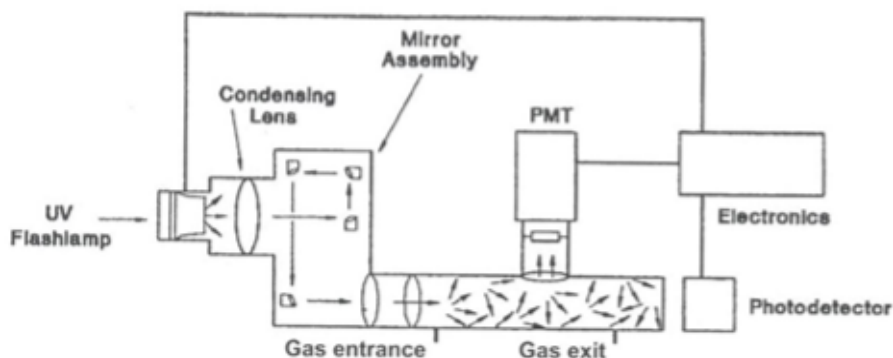


FIG. 4 PRINCIPLE OF THE UV-FLUORESCENCE DETECTOR

D-3.1.1 Principle of Operation in Seven Steps

- The sample is introduced into the furnace;
- Sulfur components oxidized to SO_2 ;
- The moisture is removed in Perma pure dryer;
- Soot and undesired particles are trapped in the filter assembly.
- In the reaction chamber, UV excites the SO_2 molecules present in the gas flow;
- The fluorescence light is detected during relaxation of the decaying SO_2^* back to SO_2 by PMT resulting in a voltage signal, proportional to the sulfur amount; and
- The PC executes the data acquisition and generates a graphic display.

D-3.2 Apparatus

D-3.2.1 Sulfur Analyser Apparatus, of renowned make equipped with liquid gas introducing module, computer and operating software.

D-3.2.2 Syringe, 100 μl capacity

D-3.2.3 Graduated Flasks, 50 ml capacity

D-3.2.4 Graduated Flasks, 100 ml capacity

D-3.2.5 Micro Burette, 10 ml size

D-3.3 Reagents

D-3.3.1 Argon Gas, high purity grade

D-3.3.2 Oxygen Gas, high purity grade

D-3.3.3 Thiophene, AR grade

D-3.3.4 Iso – Octane (2, 2, 4 Trimethyl Pentane)

D-3.4 Preparation of Solutions

All necessary solutions required for analysis are described below.

D-3.4.1 Sulfur Standards**D-3.4.1.1 Sulfur stock solution 1000 mg/l**

To make a stock solution of sulfur dissolve 0.265 gm of thiophene in 25 ml of iso – octane (2, 2, 4 trimethyl pentane), Make up to 100 ml with iso – octane. Mix the solution thoroughly.

D-3.4.1.2 Sulfur standard solution 100 mg/l

Pipette 10 ml of sulfur stock solution (**D-3.4.1.1**) in a 100 ml measuring flask and make up to 100 ml with iso – octane. Mix the solution thoroughly.

D-3.4.1.3 Sulfur standard solution 20 mg/l

Pipette 10 ml of sulfur stock solution 100 mg/l (**D-3.4.1.2**) in a 50 ml measuring flask and make up to 50 ml with iso – octane. Mix the solution thoroughly.

D-3.5 Calibration and Analysis Procedure

Accurately take 2.5 ml, 5.0 ml, 7.5 ml and 10.0 ml sulfur standard solution 20 mg/l as prepared in (**D-3.4.1.3**) into a 50 ml measuring flask using micro burette and make up to 50 ml with iso-octane. Mix the solution thoroughly. The resulting standards will be of concentrations 1.0 ppm, 2.0 ppm, 3.0 ppm and 4.0 ppm respectively. Inject each standard solution 90 μl using syringe through Liquid gas introduction module. Calibrate the method by creating calibration curve using area obtained against concentration of standard solutions.

Inject pure methanol sample 90 μl using syringe through liquid gas introduction module, software will calculate and give results using previously stored calibration curve.

D-3.6 Calculation

$$\text{Sulfur and sulfur compounds as (S)} = \frac{\text{PPM sulfur measured}}{\text{Percent by Mass}} \times \text{Sp. Gravity} \times 10^4$$

ANNEX E

[Table 1, Item (viii)]

DETERMINATION OF FREEDOM FROM CHLORIDE AND CHLORINE COMPOUNDS

E-1 REAGENTS

E-1.1 Alcoholic Caustic Potash Solution, 0.1 N, approximately

E-1.2 Dilute Sulphuric Acid, 5 N, approximately.

E-1.3 Litmus Paper, blue.

E-1.4 Sulphur Dioxide Water, saturated solution.

E-1.5 Concentrated Nitric Acid, sp. gr 1.42 (see IS 264).

E-1.6 Silver Nitrate Solution, 4 percent (*m/v*).

E-2 PROCEDURE

E-2.1 Reflux 50 ml of the material with 10 ml of alcoholic caustic potash solution for 1 h in a 250 ml conical flask fitted with an upright condenser. Transfer

the contents to a 250 ml beaker and wash down the flask and the condenser with 50 ml water into the beaker. Drive off the alcohol on a water-bath and evaporate the solution down to about 20 ml. Cool the beaker and acidify the solution faintly with dilute sulphuric acid using litmus paper as indicator. Add a drop of sulphur dioxide water and mix the contents thoroughly. Add 1 ml of concentrated nitric acid and boil to drive off nitrous fumes, if any. Cool the solution and filter, if necessary. To the clear filtrate, add 5 ml of silver nitrate solution.

E-2.2 Carry out a blank test substituting the material by the same quantity of distilled water.

E-2.3 The material shall be considered as having satisfied the requirements prescribed in Table 1, if any opalescence produced in the test with the material is not greater than that produced in the blank test.

ANNEX F

(Clause 6.1)

SAMPLING OF METHANOL

F-1 GENERAL REQUIREMENTS OF SAMPLING

F-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

F-1.1 Samples shall be taken in a protected area with good ventilation. The samples shall be kept away from flames.

F-1.2 The sampling instrument shall be clean and dry.

F-1.3 The samples, material being sampled, sampling instrument and containers for samples shall be protected from adventitious contamination.

F-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring, or both or by rolling so as to bring all portions into uniform distribution.

F-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass containers.

F-1.6 The sample containers shall be of such a size that they are almost, but not completely, filled by the sample.

F-1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with the manufacturer's name or trademark. The month and year of manufacture of the material, the batch number (if available), and other details of sampling, such as the date of sampling and samplers name.

F-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

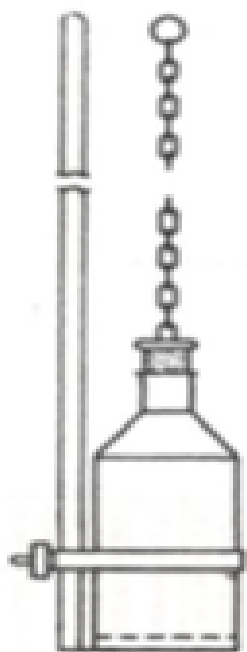
F-2 SAMPLING INSTRUMENTS

F-2.0 The following forms of sampling instruments may be used:

- a) Weighted sampling can for taking samples from various depths in large tanks, and
- b) Sampling tube.

F-2.1 Weighted Sampling Can

Of suitable capacity, 500 to 1 000 ml and of such a mass as to sink readily in the material to be sampled. It has a long chain or cord attached to permit filling at any desired level (Fig. 5). The metal used to weigh the apparatus shall be fitted externally, as irregularities in the metal are likely to contaminate the sample, if the weight is fitted internally.



All dimensions in millimeter.

FIG. 5 WEIGHING SAMPLING CAN

F-2.2 Sample Tube

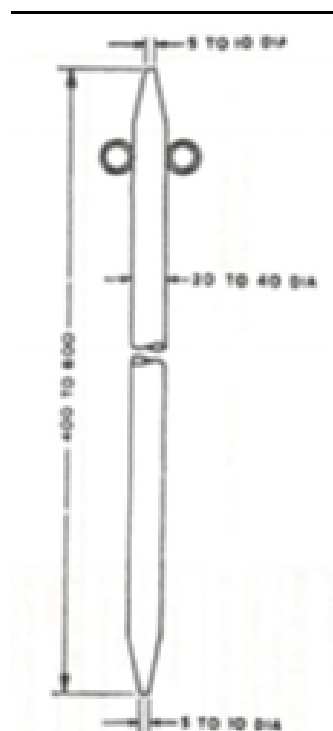
It is made of metal or thick glass and is 20 to 40 mm in diameter and 400 to 800 mm in length (see Fig. 6). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

F-2.2.1 For small containers, the size of the sampling tube may be altered suitably.

F-3 SCALE OF SAMPLING

F-3.1 Lot

In any consignment, all the containers of the same size and grade belonging to the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.



All dimensions in millimeter.

FIG. 6 SAMPLING TUBE

F-3.2 For ascertaining the conformity of the material in a lot to the requirements of this specification, tests shall be carried out for each lot separately. For this purpose, the number of containers to be selected from a lot shall be in accordance with Table 3.

Table 3 Scale of Sampling

(Clause F-3.2)

No. of Containers in the Lot	No. of Containers To be Selected
N	n
(1)	(2)
Up to 25	3
26 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

F 3.2.1 The containers shall be chosen random from the lot. To ensure the randomness of selection, procedure given in IS 4905 may be followed.

F-4 TEST SAMPLES AND REFEREE SAMPLES

F-4.1 Preparation of Sets of Samples for Testing Individually

Draw with an appropriate sampling instrument (F-2), equal portions of the material from different parts of each container selected as in F-3.2 and freshly

opened. Mix these small portions of the material from the same container to obtain a sample, representative of the container. Keep these representative samples from different containers separately. From each representative sample, draw three equal portions of the material, each sufficient for carrying out the intended tests and transfer them into thoroughly cleaned and dry sample containers. Seal the sample containers air-tight. Thus three sets of test samples are obtained such that each set has a test sample from each selected container. Send one each of these sets to the purchaser and the supplier. Reserve the third set as referee sample bearing the seals of the purchaser and the supplier. Keep the referee sample at a place agreed to between the purchaser and the supplier.

F-4.2 Preparation of Composite Samples

Draw, with an appropriate sampling instrument (*see* F-2), equal portions of material from each container selected as in F-3.2 and mix them together thoroughly to constitute a single composite sample. Divide this composite sample into three parts, each sufficient for carrying out the intended tests and transfer them to thoroughly cleaned and dry sample containers. Send one each of these to the purchaser and the supplier. Reserve the third composite sample as referee sample at a place agreed to between the purchaser and the supplier.

F-5 NUMBER OF TEST

F-5.1 Individual Samples

Test for distillation range, specific gravity, aldehydes and ketones (as acetone) shall be carried out individually on each of the test samples in a set prepared in F-4.1.

F-5.2 Composite Samples

Tests for the determination of the remaining characteristics of this specification shall be carried out on the composite sample prepared as in F-4.2

F-6 CRITERIA FOR CONFORMITY

F-6.1 For Composite Samples

The lot shall be declared as conforming to the requirements of this specification if each of the test results made on the composite sample (F-5.2) satisfies the corresponding requirements of this specification.

F-6.2 For Individual Samples

The lot shall be declared as conforming to this specification if for each of the characteristics tested on the individual samples according to F-5.1, the mean and the range of test results be calculated as below:

Mean (\bar{x}) = sum of the test results divided by the number of test results so added; and

Range (R) = difference between the maximum and minimum values of the test results.

The expression ($\bar{x} - 0.6 R$) and ($\bar{x} + 0.6 R$) shall then be calculated.

The lot shall be considered to conform in respect of the relevant characteristics, if ($\bar{x} + 0.6 R$) is less than the maximum specified value and ($\bar{x} - 0.6 R$) is greater than the minimum specified value. When the number of container selected is ten, it shall be divided into two groups of five each. Taking first five in one group and next five in other group, and for each group, range and its average shall be calculated. The mean range is:

$$\bar{R} = \frac{\text{Sum of the ranges of the two groups}}{2}$$

In the expression ($\bar{x} - 0.6 R$), R shall be replaced by \bar{R} to determine the conformity of the lot.

ANNEX G

[Table 1, SI No. (viii)]

DETERMINATION OF ASSAY

G-1 GENERAL

This test method is related to determination of the purity of methyl alcohol by gas chromatography and, in addition, provides a means for identification of certain known impurities. Water and acidity are determined by other methods and the results are used to normalize the chromatographic values.

G-2 APPARATUS

G-2.1 Gas Chromatograph

Any gas liquid chromatograph having a flame ionization detector can be used with following accessories and operating conditions:

CONDITIONNS:		
Column	:	10 Percent carbowax 20
Mesh size	:	80 /100
Column length	:	2 m
I.D	:	3 mm
Colum tubing material	:	Stainless steel (tubing must be non-reactive with substrate, sample and carrier gas)
Carrier gas	:	Nitrogen (minimum purity - 99.95 mole percent)
Syringe	:	10 µ
Sample size	:	0.2 µ
Electronic integrator	:	For computation of result
CONDITIONNS :		
Column	:	10 Percent carbowax 20
Mesh size	:	80/100
Column length	:	2 m
I.D	:	3 mm
Column tubing material	:	Stainless steel (tubing must be non-reactive with substrate, sample and carrier gas)

NOTE — The above gas chromatographic conditions are suggestive. However, any GC with different columns (packed/capillary having different stationary phase/length/diameter/film thickness) and different carrier gas (He, H₂ or N₂) may be used provided standardization/calibrations are done after setting up chromatographic conditions for required resolution.

G-3 IDENTIFICATION, CALIBRATION AND STANDARDIZATION

Install the column in the chromatograph. Set the conditions given above of column temperature and carrier gas flow that gives the necessary resolution of the components in the sample being analyzed. Set the conditions as such which gives a 10 percent, *Min* recorder deflection for a 0.1 percent concentration of purity at the most sensitive setting of instrument. Allow sufficient time for the instrument to reach equilibrium as indicated by stable base line. Adjust carrier gas flow rate to a constant value.

G-3.1 Identification

Determine the retention time of each component by injecting small amount either separately or in known mixture.

G-3.2 Calibration and Standardization

G-3.2.1 The response of any given detector varies from one chemical to another. Therefore, calibration is must. Calibration allows the computation of response factor, which expresses the relative response of different components to the detector.

G-3.2.2 Calibration standard is prepared containing the impurities present in methanol. During standard preparation the exact amounts of all the components

is noted. Inject 1.0 µ of calibration standard in the chromatographic column in accordance with the specified conditions.

G-3.2.3 Calculate response factor to four decimal places for all components relative to methanol using following equation:

$$RF_x = \frac{W_x \times A_m}{A_x \times W_m}$$

where

RF_x = response factor for component *x* relative to methanol;

W_x = weight of component *x*, in the calibration standard;

A_x = area of component *x*, in the calibration run;

W_m = weight of methanol in the calibration standard; and

A_m = area of methanol in the calibration standard run which is selected as reference component.

The component which is unknown in the sample is assigned the relative response factor 1.0000. It is advisable that the determination of response factors be made on the basis of duplicate analysis.

G-3.2.4 Response factor should be re-checked after any perceptible change in column or instrument's performance.

G-4 PROCEDURE

Inject 1.0 µl of sample into the chromatographic column using the same conditions as for components identification and standardization record and integrate the chromatogram using appropriate attenuation setting that provide optimum peak heights. Measure the area of all the peaks.

G-5 CALCULATION

Calculate the concentrations of sample components using the following equation:

$$C_x, \text{ percent} = \frac{F_x \times R_x}{\sum R_x \times F_x} \times (100 - C)$$

where

C_x = compound X, weight, percent;

R_x = area of compound X;

F_x = relative response factor of compound X; $\sum R_x$,
 F_x = sum of the individual component peak

area R_x multiplied by their relative response factors F_x ; and

C = sum of acidity and water content of the sample, percent by mass.

G-6 PRECISION AND BIAS**G-6.1 Repeatability**

Two results should be considered suspect if they differ by more than 0.0006.

G-6.2 Reproducibility

Two results should be considered suspect if they differ by more than 0.0027.

NOTE — Assay/purity of methanol can also be derived by subtracting sum of (acidity, moisture and impurities namely, acetone, ethanol and methyl ethyl ketone)

Percent methanol = (100 – C)

Where, C = Sum of moisture, acidity as acetic acid, ethanol, acetone and methyl ethyl ketone.

ANNEX H

(Foreword)

COMMITTEE COMPOSITION

Organic, Chemicals Alcohols and Allied Products Sectional Committee, PCD 09

<i>Organization</i>	<i>Representative(s)</i>
Chemical Engineering and Process Development Division, NCL Pune	DR C. V. RODE (Chairman)
All India Distilleries Association (AIDA), New Delhi	SHRI V. N. RAINA
BASF India Limited, Mumbai	SHRI KIRAN BHAT SHRI HEMAL BERAWALA (<i>Alternate</i>)
CDRI, Lucknow	DR SANJEEV KANOJIYA
Central Revenues Control Laboratory, Delhi	DR T. A. SREENIVASA RAO
Chemical and Petrochemicals Manufacturers Association (CPMA), New Delhi	SHRI KAMAL NANAVATY
Deepak Fertilizer, Pune	DR SATISH CHAND SAINI SHRI SURESH AMLE (<i>Alternate</i>)
Deepak Phenolics Limited, Bharuch	DR S. SAMAL SHRI N. R. SANDIPKUMAR PANCHIA (<i>Alternate</i>)
Dow-Corning India Limited, Mumbai	SHRI ABRAHAM BARRETTO. SHRI RITESH GULABANI (<i>Alternate</i>)
Indian Chemical Council, Mumbai	DR MRITUNJAY CHAUBEY SHRI J. I. SEVAK (<i>Alternate</i>)
Jubilant life Sciences Ltd, Noida	SHRI HARI MOHAN LOHANI
National Chemical Laboratory, Pune	DR UDAYA KIRAN MARELLI
UPL Limited, Mumbai	MR M. D. VACHHANI, GM (QA)
Alkyl Amines Chemicals Ltd, Mumbai	SHRI S. V. NIKUMBHE SHRI SAMEER KATDARE (<i>Alternate</i>)
All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	SHRI K. L. RAPHAEL SHRI KIRTI GAJJAR (<i>Alternate</i>)
Godavari Biorefineries Ltd, Mumbai	SHRI SHANUL LAXMANRAO PAGAR MS WANI A. J. (<i>Alternate</i>)
Gujarat Narmada Valley Fertilizers Company Limited, Gujrat	DR M. J. KAPADIA SHRI P. R. DESAI (<i>Alternate</i>)
Hindustan organic chemicals Ltd (HOCL), Mumbai	SHRI DELEEP KUMAR K. SHRI V. MOHAN (<i>Alternate</i>)
India Glycols Limited, Uttarakhand	SHRI S. R. SONI SHRI ALOK SINGHAL (<i>Alternate</i>)
Laxmi Organic Industries, Mumbai	SHRI J. P. SURYAVANSHI DR VIJAY S. MISHRA (<i>Alternate</i>)
Ministry of Chemicals & Fertilizers, New Delhi	SHRI O. P. SHARMA SHRI VARUN SINGH POONIA (<i>Alternate</i>)
National Test House, Ghaziabad	SHRI DEBASHIS SAHA DR GOPAL KRISHAN (<i>Alternate</i>)
Reliance India Limited, Mumbai	SHRI K. K. SREERAMACHANDRAN SHRI VASANT WARKE (<i>Alternate</i>)
BIS Director General, New Delhi	SHRI N. K. BANSAL, SCIENTIST, 'F' AND HEAD (PCD), [REPRESENTING DIRECTOR GENERAL (<i>Ex-officio</i>)]

*Member Secretary*SHRI CHANDRAKESH SINGH
SCIENTIST 'D' (PCD), BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act, 2016* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc No.: PCD 09 (15204).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002

Telephones: 2323 0131, 2323 3375, 2323 9402

Website: www.bis.gov.in

Regional Offices:

Telephones

Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	{ 2323 7617 2323 3841
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi KOLKATA 700054	{ 2337 8499, 2337 8561 2337 8626, 2337 9120
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg CHANDIGARH 160019	{ 265 0206 265 0290
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	{ 2254 1216, 2254 1442 2254 2519, 2254 2315
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400093	{ 2832 9295, 2832 7858 2832 7891, 2832 7892

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. COIMBATORE.
DEHRADUN. DURGAPUR. FARIDABAD. GHAZIABAD. GUWAHATI.
HYDERABAD. JAIPUR. JAMMU. JAMSHEDPUR. KOCHI. LUCKNOW.
NAGPUR. PARWANOO. PATNA. PUNE. RAIPUR. RAJKOT. VISAKHAPATNAM.

Published by BIS, New Delhi